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EXPERIMENTAL EVALUATION OF PEAK HEIGHT APPROXIMATION FOR X-RAY --ETC(U)
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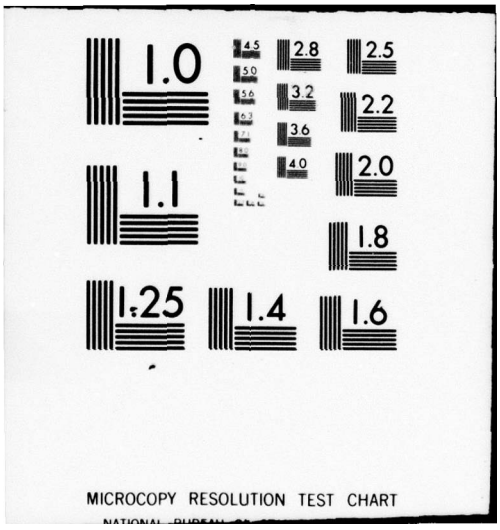
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EXPERIMENTAL EVALUATION OF PEAK HEIGHT APPROXIMATION FOR X-RAY DIFFRACTED INTEGRATED INTENSITY METHOD

April 1979

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
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ABSTRACT

A method for characterizing an unresolved characteristic X-ray K_α diffracted powder peak with respect to integrated intensity and Bragg angle has been tested. The method assumes the upper half of the diffracted peak to be Gaussian and the bottom half a Cauchy functional shape to match the experimentally measured diffraction peak. The peak height is used to compute the integrated intensity using calculated relationships. The integrated intensities of the CuK_α (111) and (200) X-ray diffracted peak doublets were measured from silicon powders employing variations in beam and receiving slit widths on commercial X-ray units and compared with the calculated intensities. This method offers promise as a fast and accurate technique as demonstrated by its ability to measure X-ray intensities to a precision of several percent under selective experimental conditions.

INTRODUCTION

The mathematical description of an X-ray peak, diffracted from a powder or polycrystalline material, using physically meaningful parameters has been of interest for many years. With the popularity of computers, this need to characterize a diffraction peak has intensified. In fact, the movement toward automated equipment and analysis of data with large, high-speed computers has increased the demands for more accurate representation of the X-ray diffractogram in terms of intensity versus Bragg angle histograms or more desirable mathematical functions.

The first intensive effort to accurately describe experimental diffracted peak profiles of an X-ray diffraction powder scan for quantitative phase analysis was performed by Smith.[1,2] Smith generated a theoretical powder diffraction scan using known physical characteristics, i.e., unit cell size, wavelength, etc., assuming either Gaussian or Cauchy functions in an attempt to describe the diffracted peaks. Both functions were found to be inadequate, with the Gaussian function dropping off too sharply from the peak maximum to background, while the Cauchy function described the "tails" well enough, but was too sharp to adequately fit the upper half of the peak. Such an approach remained qualitative or, at best, a crude quantitative tool in keeping with the operating mode of the ASTM powder diffraction system which identifies crystalline phase by approximating their X-ray diffracted intensities.

In another area, X-ray diffraction analysis was used to determine the detailed arrangement of atoms in crystals. X-ray powder techniques were introduced as a slow and incomplete method of crystal structure analysis, but yielded to the almost exclusive employment of single crystals in deducing the position of atoms.

In fact, the feasibility of deducing crystal structures from powders was demonstrated with the introduction of the powder method by Debye and Sherrer[3] using LiF powder and independently by Hull[4] on iron powder. Shortly thereafter, structures of simple materials, i.e., elements, were determined and in the years that followed crystal structure determinations were usually performed only on powders where single crystals were not available (i.e., γ-brass[5] and α-Mn[6]). For supplemental review see Peiser et al.[7] As the factors contributing to the X-ray diffracted powder peak intensity were identified and characterized, i.e., thermal, absorption, etc., the application of the powder diffraction method to crystal structural refinement became more desirable. At the present time, under favorable conditions, the practical limit of precision for measuring integrated intensities in powders is approximately 1%.[8]

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2. SMITH, D. K. *Computer Simulation of X-ray Diffractometer Traces.* Norelco Reporter, v. XV, no. 2, 1968, p. 57-76.
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With the advent of Rietveld's computer procedure for describing a complete diffraction pattern in finite 2θ steps[9,10] together with the subsequent modification of such a pattern by varying physical parameters, an improved method for structural refinement was created. Since this approach involved neutron diffraction, Gaussian functions were adequate to describe the diffracted peaks. Although Rietveld's method was readily adapted to structural refinement using neutron diffraction and was generally accepted as an advancement in diffraction analysis, it was not until 1975 that the crystallographic community began thinking of applying this technique to X-ray powder patterns. For a complete review of this development, see treatise by Young et al.[11]

A key problem which persists is how to describe the instrumental diffracted profile and therefore the observed diffracted characteristic peak with subsequent combinations of K_α doublets and mixed overlapping peaks. Many attempts have been made at finding a "true" function to fit the observed diffracted peak; however, a practical solution has yet to be found. The reason is twofold. First, many investigators since Smith have tried to find one function that duplicates the X-ray powder peak, finding similar failures with either Gaussian, Cauchy (pure or modified), or Lorentz functions. These results are summarized by Khattak and Cox,[12] who claim to have found a reasonable fit using a cross between a modified and a pure Lorentz function. Secondly, the employment of a composite of three or four[13] or seven[14] Lorentzians adequately describe an X-ray diffracted peak but require the use of untenable computer programs for pattern-fitting crystal structure refinements, or for quantitative phase analyses where many compounds or mixtures are involved. Similar problems arise using convolution techniques involving multiple functions or angle-dependent Fourier coefficients.

The intent of this paper is to experimentally test, in part, a simple but effective alternative to the previously mentioned attempts[15] to characterize an X-ray diffracted peak. Simply stated, the essence of this method is to fit a Gaussian function to the top half and a Cauchy function to the bottom half of the diffraction peak. An application of this procedure to an X-ray diffraction scan of a mixture of silicon and α and β silicon nitride is reviewed[16] in which the integrated intensity is approximated by the peak height for one set of experimental conditions (i.e., a single beam and receiving slit). The degree of correlation of the X-ray peak heights to compositional levels is taken as confirmation of the validity of the proposed method. Another check on the accuracy of

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10. RIETVELD, H. M. *A Profile Refinement Method for Nuclear and Magnetic Structures.* J. Appl. Cryst., v. 2, 1969, p. 65-71.
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the curve fit is the improvement in the accuracy of lattice constant measurements of silicon using the peak positions of the K_{α_1} and K_{α_2} unresolved doublets convoluted from the Gaussian-Cauchy curves.[17]

The final endorsement of this method is the experimental evidence presented in this study, using silicon powder. In this case the measurement of integrated intensities are performed using variations in slit systems for different commercially available X-ray units for testing the effectiveness of the peak height approximation as well as the peak shape-fitting technique.

PROCEDURE

The relationship between the X-ray diffracted peak height y_T and the integrated intensity of the peak was theoretically developed[15] for one set of experimental conditions. The mathematical functions that approximate the K_α characteristic diffraction peaks are (1) a Gaussian curve to match the upper half and (2) a Cauchy function to fit the bottom half, of the form:

$$y = 1/[1 + k^2 (x_1 - a)^2] \qquad (1)$$

where x_1 is a linear function of the Bragg angle, varying from 0 to 2 for the full separation of the K_{α_1} and the K_{α_2} peaks, a represents the peak separation and equals $(2.36d\theta/\omega_{h\frac{1}{2}})$, and $\omega_{h\frac{1}{2}}$ = peak width at half height for the resolved K_{α_1} diffraction peak.

The low angle side of a CuK_{α_1} (531) reflection of silicon powder, scanned with a very narrow receiving slit (0.01°), matched Equation 1 for a value of $k^2 = 0.7234$.

These mathematical functions were used to generate constant intensity curves of y_T versus 2θ angles for different levels of $\omega_{h\frac{1}{2}}$ (see Figure 1). In this case $\omega_{h\frac{1}{2}}$ is assumed to be constant over the 2θ range considered. One major advantage of this analysis was the ability to determine this change in peak height for an unresolved K_α doublet which is usually what is detected in practice. A relationship[18] whereby $\omega_{h\frac{1}{2}}$ can be abstracted from the half width of the K_α doublet $\omega_{T\frac{1}{2}}$ was worked out[15] and given in Figure 2. Therefore, the usual procedure is to measure $\omega_{T\frac{1}{2}}$ for two (in the case of Si using CuK_α radiation) or more peaks, read the respective values of $\omega_{h\frac{1}{2}}$ from Figure 2, and obtain the decrease in peak height for the corresponding angle 2θ from Figure 1.

In this study this procedure is tested for different combinations of beam and receiving slits on three commercial X-ray systems using the change in height of the silicon (111) peak at 28.5°2θ, and the (200) peak found at 47.4°2θ.

Diffraction scans of the two silicon peaks were performed for combinations of beam and receiving slits, and their integrated intensities measured by planimetering the diffractograms. The criterion for the accuracy involves first computing the change in peak height from the (111) to (200) reflection of silicon

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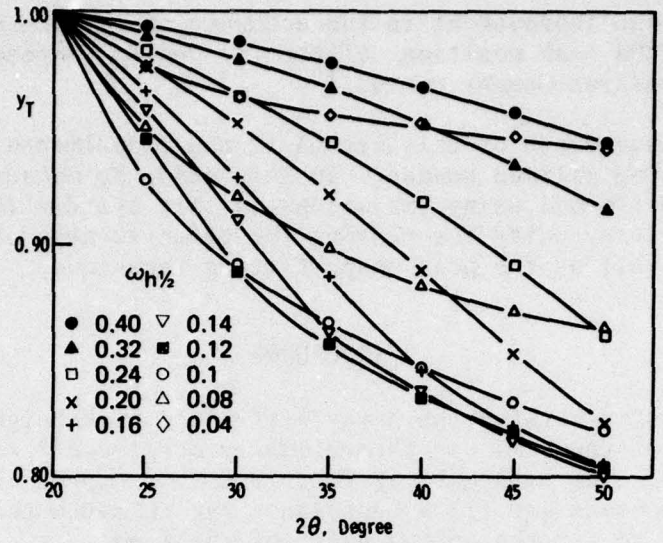


Figure 1. y_T versus 2θ curves calculated for constant levels of $\omega_{h½}$ from 0.04 to 0.40.

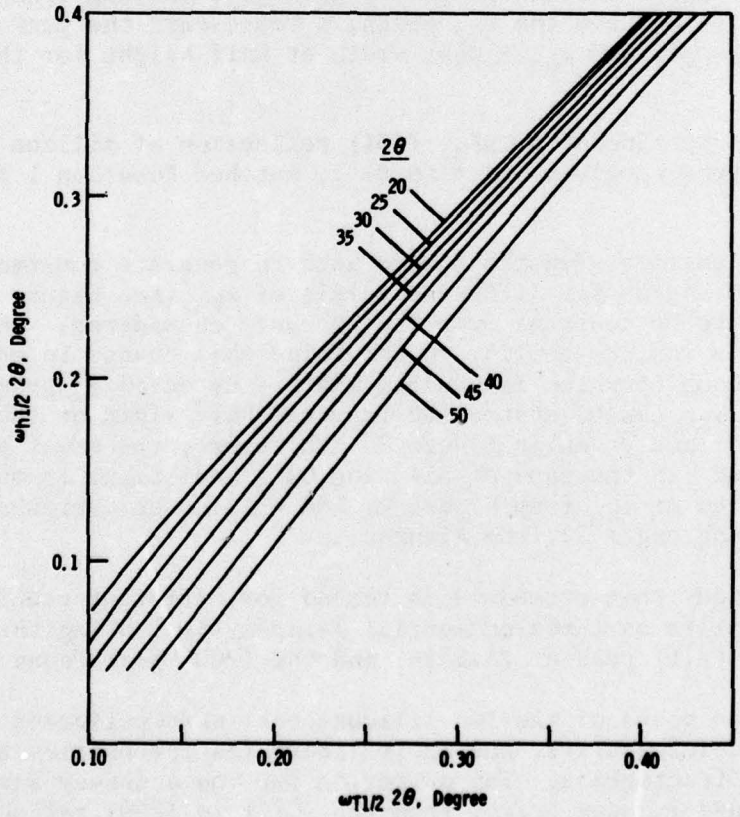


Figure 2. $\omega_{h½}$ versus $\omega_{T½}$ curves calculated for various levels of 2θ from 20° to 50° assuming Gaussian and Cauchy peak distributions.

$\Delta y_{T\ CALC}$ assuming constant intensity, using Figures 1 and 2. Then, the ratio of the measured integrated intensities, I(111)/I(200), was multiplied by the inverse ratio of the measured peak heights, $y_T(200)/y_T(111)$, to yield the observed reduction in peak height, $\Delta y_{T\ OBS}$. The difference, $\Delta y_{T\ CALC} - \Delta y_{T\ OBS}$, in percent, is called % deviation $_c$, and is taken as the measure of accuracy for this method. (The subscript c denotes that a constant value of $\omega_{h\frac{1}{2}}$ is assumed.)

As will be seen for the Norelco X-ray system, the condition that $\omega_{h\frac{1}{2}}$ remain constant is not met for case 2. In this case, therefore, $\Delta y_{T\ OBS}$ is multiplied by $\omega_{T\frac{1}{2}}(200)/\omega_{T\frac{1}{2}}(200)'$ where $\omega_{T\frac{1}{2}}(200)'$ is the apparent doublet peak width, at half height, read from Figure 2 starting with the observed value of $\omega_{T\frac{1}{2}}(111)$. Or,

$$\Delta y'_{T\ OBS} = \Delta y_{T\ OBS} \times \omega_{T\frac{1}{2}}(200)/\omega_{T\frac{1}{2}}(200)' \qquad (2)$$

and % deviation $_v = \Delta y_{T\ CALC} - \Delta y'_{T\ OBS}$. (The subscript v indicates that $\omega_{h\frac{1}{2}}$ is variable.) The justification for calculating $\Delta y'_{T\ OBS}$ assumes that the convolution of the instrumental functions do not significantly affect the shape of the diffracted X-ray peak with a variation in the slit systems, e.g., the diffracted X-ray peak will remain essentially Gaussian with the convolution of a Gaussian instrumental function.[15]

EXPERIMENTAL

The silicon powder examined was less than 400 mesh particle size. Filtered CuK_α (line focus) was selected as the X-radiation. The three X-ray diffraction systems that yielded the data for this study are listed together with the experimental conditions:

A. General Electric XRD-5

Tube - fine focus (0.4 mm × 12.5 mm)
Take-off angle - 2°
Medium resolution soller slits

Slits, degrees -

Beam	Receiving
3	0.02, 0.05, 0.1, 0.2
1	0.01, 0.02, 0.05, 0.1, 0.2, 0.3, 0.4
0.4	0.02, 0.05, 0.1, 0.2

B. Siemens Crystalloflex IV (type F)

Tube - oil immersed X-ray tube (0.4 mm × 8 mm)
Take-off angle - 3°
Medium resolution soller slits

Slits, degrees -

Beam	Receiving
1/2, 1, 2	0.05, 0.1, 0.2

Monochromator - Eastern Scientific (Pyrolytic Graphite Curved Crystal)

C. Norelco (Vertical diffractometer with standard soller slits)

Standard tube
Take-off angle - approximately 4°

Slits, degrees -

	Beam	Receiving
Case 1	1/2, 1, 2	0.006"
		Scatter - 1/12, 1/6, 1/4, 1/2, 1, 3
Case 2	1/4, 1/2, 1, 2	0.026, 0.068, 0.154

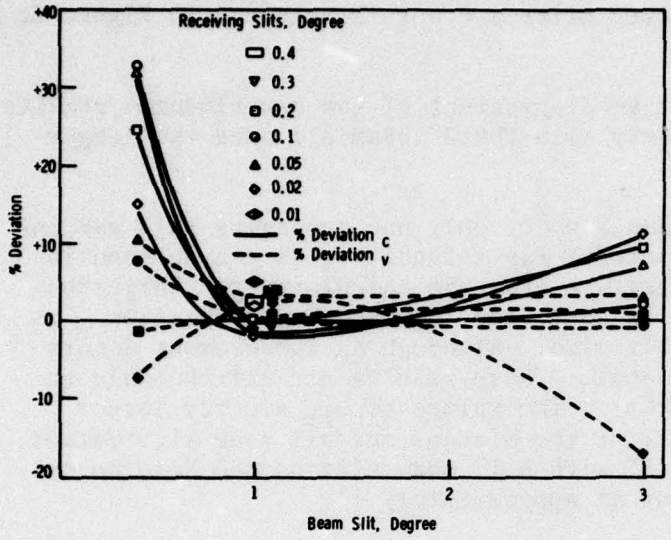
RESULTS

Values of $\Delta y_{T\ OBS}$ and $\Delta y'_{T\ OBS}$ were measured and the % deviation $_c$ and % deviation $_v$ calculated and plotted in Figure 3 for various configurations of beam and receiving slits.

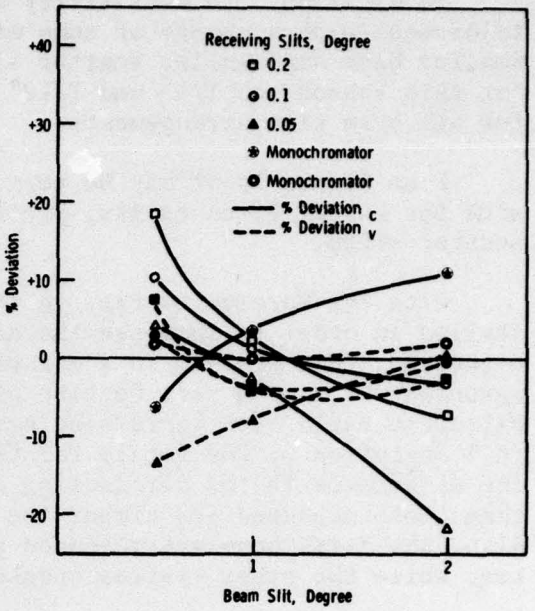
The results for the General Electric system, seen in Figure 3a, are not surprising. Since the functions chosen to match the (531) reflection of Si were obtained with a beam slit of 1°, the present experimental results would be expected to possess a good fit with the same beam slit. These results do not change in going from % deviation $_c$ to % deviation $_v$, confirming the existence of a constancy in $\omega_{h\frac{1}{2}}$ between the two reflections investigated. Some departure from the ideal case (i.e., % deviation $_c$ = 0) exists with the 3° beam slit, but improves in going to % deviation $_v$, except for a very narrow receiving slit. As expected, the increase in % deviation $_c$ is high for a 0.4° beam slit, with some improvement in correcting for the change in peak width, but also increasing the scatter in % deviation $_v$ with a variation of the receiving slit.

The plots for the Siemens system (Figure 3b) show a similar behavior for a small beam slit (½°). For a beam slit of 1° with 0.1° and 0.2° receiving slits, the behavior of % deviation $_c$ is excellent, with a decrease of approximately 5% in the value of % deviation $_v$. A reversal in this behavior occurs for a beam slit of 2°. In this case, the % deviation $_c$ values are too low, and increase to a level in fairly good agreement with the calculated values, or % deviation $_v \approx 0$. The monochromator yields an interesting situation. The value of % deviation $_c$ is within the exterimental limits for a beam slit of 1°, but is unreasonably high at 2°, while the values of % deviation $_v$ are excellent for all the beam slits.

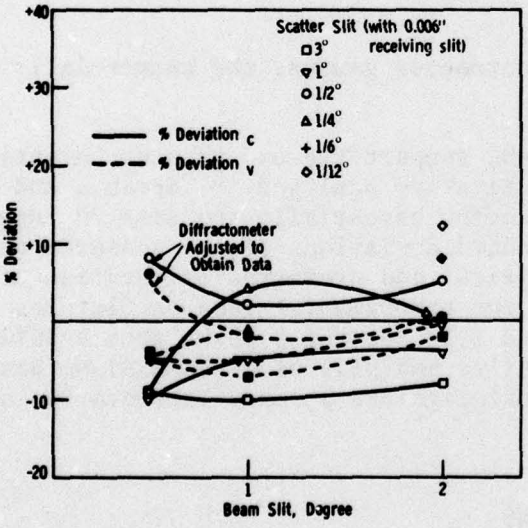
The Norelco system warrants some consideration due to the special arrangement of the slit geometry. In the conventional case (case 1), a scatter slit, which probably should be termed a defining slit, provides a high degree of resolution of the diffracted beam. In fact, the CuK_{α_1} and CuK_{α_2} silicon peaks are resolved to a higher degree than with either of the other two X-ray systems. This condition, however, is achieved at the expense of a loss in integrated intensity of the diffracted peak. With the slit arrangement involving a 2° beam slit and removing the 0.006" receiving slit (proceeding from case 1, with a 0.006" receiving slit and a 1° scatter slit to case 2 with a 0.154° receiving slit), the integrated intensity is increased by a factor of eight times.



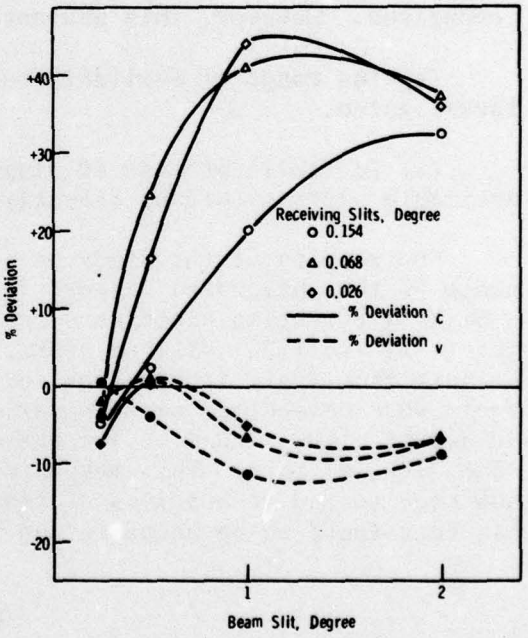
a. General Electric XRD-5 X-ray System.



b. Siemens Crystalloflex IV Type F X-ray System.



c. Norelco Vertical Diffractometer X-ray System, with a conventional slit system.



d. Norelco Vertical Diffractometer X-ray System, with one receiving slit.

Figure 3. Percent deviation of experimental X-ray diffracted (111), (200) silicon powder intensities from theoretical values versus slit configurations.

In addition, the sensitivity of the slit alignment for case 1 demanded readjustment with a change of some of the scatter slits, particularly with the smaller beam and smaller scatter slits, thus affecting diffracting conditions. For this reason the 1/6° and 1/12° scatter slits are not illustrated in Figure 3c for all beam slit arrangements.

From Figure 3c it may be seen that good agreement of the experimental results with the calculations exists, particularly with the 2° beam slit and the larger scatter slits.

With the Norelco system, in the case 2 mode, only one receiving slit was installed in order to increase the diffracted X-ray intensity. As can be seen in Figure 3d, this results in a marked departure from the calculations. While the agreement at a beam slit setting of ¼° is good, the increase in % deviation $_c$ is extremely rapid with increasing beam slit size. Although an improvement occurs in % deviation $_\gamma$, the fit is far from ideal. These results are attributable to the difference in the diffracting geometry. The values of $\omega_{T\frac{1}{2}}$ are far larger than those measured for either the G. E. or the Siemens for all beam slit values. Also, the X-ray beam was measured at 1.7° with a 1° beam slit on the Norelco system, while the other systems checked out at approximately 1°.

A 3° beam slit was not tested with the Norelco system since the 2° beam slit already covered up to 7/8" of the sample at the CuK_α (111) 2θ position.

Perhaps it would have been more appropriate to use $\omega_{T\frac{1}{2}}$ as a parameter for comparison. However, this was not done for two reasons.

(1) The range of available beam slits is not great enough for all systems investigated.

(2) To facilitate use of simpler diffractometer gauges, the commercially available slits should be directly involved.

The results of the study on α and β Si_3N_4 support the use of peak heights in place of the integrated intensities for quantitative analyses.[16] Seven α and four β Si_3N_4 diffraction peaks were selected as having essentially the same 2θ range as the (111) and (200) silicon peaks. The standard deviations of the measured peak heights from their true values, after statistical and preferred orientation effects were corrected, were approximately 5% for experimental runs on mixtures of 80%α-20%β Si_3N_4, and 6.6% for the α peaks and 2.3% for the β peaks from a 55%α-45%β Si_3N_4 mixture. This method of quantitative analysis of α and β Si_3N_4 has now been tested on hundreds of samples by investigators at many laboratories and has been found to be accurate and reliable.

CONCLUSIONS

This study has given further justification to the use of a simplified method of approximating the shape of an X-ray diffraction peak and approximating its integrated intensity using the peak height and breadth. The prior verification of

this technique has been reported[16] in its use in the quantitative analysis of Si_3N_4 and in the improvement in the accuracy of measured lattice constant values of silicon using this convolution technique.[17]

The advantages of using this procedure are numerous. A few of the more important features are:

(1) Efficiency - Savings in operator and computer time, speed of obtaining intensities where many intensity values are necessary.

(2) Simplicity - The X-ray operator need not know the physical factors affecting the peak shape.

(3) Search Systems Application - Thousands of ASTM Powder Diffraction file cards contain X-ray diffraction intensities based on uncorrected peak heights. With this system of approximating intensities, better intensity data correlation with the ASTM cards may be possible.

(4) Utility - X-ray diffraction peak profile fitting can be accomplished with a small computer, making applications accessible to more investigators.

Before assurance can be given that the procedure outlined can be freely used to approximate X-ray diffracted integrated intensities the following suggestions are in order:

(1) This procedure should be tested using materials other than silicon. The only other materials tested to date have been α and β Si_3N_4, copper, and Al_2Cu.[15,16] More tests should be made under different experimental conditions on more materials.

(2) Testing on the Gaussian-Cauchy curve fitting should be performed and its effect on the accuracy of the resulting peak heights determined. In fact, before the procedure is applied, the shape of the characteristic line should be matched to the Gaussian-Cauchy functions.

(3) Particular care should be exercised when trying to apply the suggested method to an X-ray system involving a variation from the standard diffracted beam geometry, as in case 2. More exploratory work is necessary before a dependable procedure can be effected.

(4) Notwithstanding the fact that several areas remain to be investigated the results given in this report can be applied, employing selected slit arrangements to efficiently yield integrated intensities from X-ray diffracted peak heights.

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Army Materials and Mechanics Research Center,
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EXPERIMENTAL EVALUATION OF PEAK HEIGHT
APPROXIMATION FOR X-RAY DIFFRACTED
INTEGRATED INTENSITY METHOD -
Charles P. Gazzara

Technical Report AMMRC TR 79-22, April 1979, 11 pp -
illus, D/A Project 1L161102AH42,
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X-ray intensity
X-ray peak height
X-ray analysis

A method for characterizing an unresolved characteristic X-ray K_α diffracted powder peak with respect to integrated intensity and Bragg angle has been tested. The method assumes the upper half of the diffracted peak to be Gaussian and the bottom half a Cauchy functional shape to match the experimentally measured diffraction peak. The peak height is used to compute the integrated intensity using calculated relationships. The integrated intensities of the CuK_α (111) and (200) X-ray diffracted peak doublets were measured from silicon powders employing variations in beam and receiving slit widths on commercial X-ray units and compared with the calculated intensities. This method offers promise as a fast and accurate technique as demonstrated by its ability to measure X-ray intensities to a precision of several percent under selective experimental conditions.

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